

# ***SURFACE MODIFICATION PROCESS ON METAL DENTURES, PRODUCTS PRODUCED THEREBY, AND THE INCORPORATED SYSTEM THEREOF***

## **BACKGROUND OF THE INVENTION**

The requirement of dentures is meaningless to detail here. It is also human history to seek better dentures to keep the human healthy life by increasing the pleasure of the table.

There are some materials to compose the dentures as plastics, ceramics, metals and others. Metal or partial metal dentures are dentures made with metal materials, which is said best materials now. The metal materials are roughly classified into three categories of Titanium System Alloys, Cobalt System Alloys and Precious System Alloys.

As the recent trend, the requirement of Titanium System Alloys are in majority, on the other hand Precious System Alloys are in minor due to its deformation phenomena or heavier weight.

The features of metal and/or partial metal dentures are well fitness in the mouth, immediate reaction of hot foods in the mouth due to its high thermal conductivity and its lightweight in case of Titanium System Alloys.

On the other hand, to fabricate the metal and/or partial metal dentures requires the long years experience of dental technicians and their well-performed skills. Therefore it takes much time to fabricate the metal and/or partial metal dentures, and it usually highly costs.

To fabricate the metal and/or partial metal dentures, there are plenty of steps as alloys melting first, then casting in the mold and finishing the surface with handy diamond small abrasive tools.

After de-molding, the cast metal and/or partial metal dentures are finished by a well-experienced dental technician with handy tools, therefore the technician is able to finish one metal and/or partial metal denture in two or three hours or in half day in longest. Moreover, the surface finishing in the process are to remove the cast skin layer and fit the mouth shape from the metal and/or partial metal dentures, then the surface are to be corroded by the acid in the stomach to change the color from metallic to black.

## **SUMMARY OF THE INVENTION**

In the mentioned invention, electron beams in pulsed repetition bombarded onto the total surface of the metal and/or partial metal dentures. The surface of the metal and/or partial metal dentures as its after the cast were melted and turned into amorphous surface. This eliminates the time and cost of the cast metal and/or partial metal dentures with handy diamond tools by a well-experienced dental technician. Due to amorphous metal surface, the electron beam treated surface shows 2 to 5 times stronger resistance against accelerated hydrochloride acid solution. The invention contributes to reduce the time to provide the metal and/or partial metal dentures with several minutes from hours fabrication; moreover, its acid corrosion resistance is dramatically increased to perform for

long years use.

## BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 shows the schematic general assembly of pulsed electron beam system, which was used to modify the surface of the metal and/or partial metal dentures.

Fig. 2 indicates typical operating condition as accelerated voltage and beam current.

Fig. 3 shows the operation data in the pulsed electron beam system. In the chamber, there exists partial Argon gas in vacuum chamber and the beam irradiated onto the metal and/or partial metal dentures

Fig. 4 shows the visible light reflection ratio from the metal and/or partial metal dentures. To make clear the increase of reflection ratio, the inventors apply the silver metal against the light beam wavelength from 300nm to 800nm. Comparing to the untreated sample, the light beam reflection ratio was remarkably increased after the pulsed electron beam bombardment.

Fig. 5 shows the X-ray diffraction for samples before and after the pulsed electron irradiation, respectively. X-ray diffraction result of the sample before irradiation shows the peak points of Titanium Metal. On the other hand, the X ray diffraction after irradiation shows some peak of re-crystallized Titanium Metal. This indicates that the surface of the metal and/or partial metal dentures is turned into semi-amorphous condition.

Fig. 6 shows the results of anodic polarization of before and after pulsed electron beam irradiation onto pure Titanium metal dentures. In this case, the counter electrode material is platinum and the solution was 1 Normal Hydrochloride water solution.

## DETAILED DESCRIPTION OF THE INVENTION

The schematic assembly of Pulsed Electron Beam System is shown in the Fig. 1. The vacuum chamber is consisted with stainless steel. The original vacuum degree is about  $10^{-2}$  Pa and Argon gas is partially filled. The cumulated energy is discharged from the cathode to the collector through double layer between the cathode plasma and the anode plasma column. For formation of the plasma column, a high current reflected (Penning) discharge in Ar working gas is applied.

The said pulsed electron beam system has anode with shape of ring or topological similar to it (for example rectangular, polygonal ring structure etc.), which is installed in such position where the axis of the ring is parallel to the beam transportation path. This position has benefit of preventing a decrease of beam current caused by collision of electrons on the anode surface.

Detailed processes of electron beam treatment are described as follows. After setting the sample on the sample holder and close the chamber lid, the chamber 1 is evacuated from atmospheric pressure until the initial background pressure by using scroll pump 2 for roughing vacuum and turbo molecular pump 3 for high vacuum evacuation. Then, Ar gas is filled in and its pressure is adjusted up to a certain pressure between  $0.5-3 \times 10^{-1}$  Pa by using flow control valve 4. After reaching the setting pressure, a pulse guide magnetic field with strength up to 4.4 kOe is created by

two external solenoids 5. In Fig.2 it is shown a typical current pulse of solenoid 14. During the generation of magnetic field, a positive pulse high voltage (about 5 kV) with anode pulse current 15 is applied to a ring-shaped anode 6 to obtain an intensive anode-plasma 7 by a reflected Penning discharge method. As another method, anode plasma might be produced simply by arc discharge, but the method would wear out the electrode, which may lead to chamber contamination. Moreover, comparing the consuming current, the arc discharge needs higher current than the present method.

After obtaining high current stage of anode plasma current (generally 20-50  $\mu$ s after applying voltage on the anode), a negative accelerating voltage pulse 16 with amplitude below 50 kV and rise time of 5-10 ns is applied to the electron gun cathode 8. An intensive electric field between the cathode and the near-cathode layer of the ionic space charge, can reach to considerable values up to 500 kV/cm and is enough to initiate an explosive electron emission for formation of a dense cathode-plasma 9. The accelerating voltage applied on the anode and cathode is concentrated in a double layer 10 created between the cathode and anode plasmas, where the high current electron beam 11 is formed. The electron beam current density in the double layer is related to the anode plasma ion current density by Langmuir law.

The electron beam accelerated in the double layer is transported through the anode plasma to the beam collector, on which the samples is placed. Owing to the ionic space charge by the anode plasma ions in the present system, the high current electron beam can be more stable compared to beams in vacuum. As usually occurred in vacuum-electron beam systems, due to repulsive Coulomb force between the electrons, the electron beam may be scattered to cause a decrease of beam current. In the present electron beam system, the decrease of the beam current can be suppressed by the existence of ions in the anode plasma.

### EXAMPLE 1

The metal and/or partial metal dentures 12 are set on the rotating sample holder 13. The beam energy density was 3 J/cm<sup>2</sup>. After the electron beam bombardment with 5 pulses repetition, the as-cast metal and/or partial metal dentures show metallic glittering surface and amorphous layer. This indicates that the hand polishing with diamond tools are not required. In case that the rotating metal holder is thermally insulated with rock wool, the surface is melting down further than the expected.

### EXAMPLE 2

Reducing the accelerated voltage from 30, 27, 25 kV and the remained partial gas pressure was increased as the irradiated energy is reduced. When the irradiated energy is lower than 0.1 J/cm<sup>2</sup> on the metal and/or partial metal dentures, any surface reaction was not observed even in the case of low melting alloys as Magnesium, Titanium, and high thermal conductive metal as Aluminum.

### EXAMPLE 3

In case the pulse repetition exceeds 100 with energy density of 3 J/cm<sup>2</sup>, the metal and/or partial

metal dentures are deformed and it lost the fitness to mouth. On the other hand, no surface modification was observed with pulse repetition over 30 with energy density of  $0.1 \text{ J/cm}^2$ .

#### EXAMPLE 4

After the electron beam irradiation with condition stated in Example 1 (i.e. energy density is  $3 \text{ J/cm}^2$ , pulse duration is  $1.54 \text{ s}$  and pulse repetition is 5 times), the surface of the metal and/or partial metal dentures shows the equivalent reflectance as the dental specialist polished with diamond hand tools. To indicate the reflection ratio numerically, we applied spectroscopy analysis against reflected beam with the beam length range of 300 to 800 nm as the result shown in Fig. 4. At almost maximum peak wavelength (i.e. 535 nm), the reflection rate after electron beam irradiation was 71% from the metal and/or partial metal dentures. On the other hand, the metal and/or partial metal dentures before the electron beam irradiation shows 35.5%. In the case, the materials used for the metal and/or partial metal dentures are Ag-Pd-Au system alloy.

#### EXAMPLE 5

To the metal and/or partial metal dentures made with Titanium system alloys, X-ray diffraction is applied to determine the surface crystal phase. The result is shown in Fig. 5 for the before and after electron beam irradiation on the metal and/or partial metal dentures. For the before electron beam irradiation metal dentures, the titanium peaks are observed, on the other hand, the titanium peaks could not be found in the dentures of the after electron beams irradiation except some peaks from re-crystallization. This shows that the surface of metal and/or partial metal dentures after the electron beam irradiation turned into amorphous.

#### EXAMPLE 6

To determine how the corrosion resistance was modified through the electron beam irradiation, Anodic polarity method was applied on pure Titanium as the result is shown in Fig. 6. Applying Tafel equation, the corrosion electric current was compared between the after electron beam irradiated and the before electron beam irradiated. This was measured in 1N HCl water solution. As the result, the corrosion electric current show  $0.03 \mu\text{A/cm}^2$  and  $0.01 \mu\text{A/cm}^2$ , respectively. This means that the metal and/or partial metal dentures after electron beam irradiation have three times stronger resistance than the non-irradiated metal and/or partial metal dentures.

#### EXAMPLE 7

The amplitude of the applied voltage to the accelerating gap exceeds than 50 kV. After the pulse repetition exceeds 3, the metal and/or partial metal dentures were thermal deformed though it indicated mirror and amorphous surface.

#### EXAMPLE 8

The electron beam irradiation was done in continued mode. In the case the heat cycle diffusion,

heating and rapid cooling, cannot be done well and homogeneous amorphous surface could not be obtained.

#### EXAMPLE 9

Pulse duration was varied and in case the duration was shorter than  $0.5 \mu\text{s}$ , the microcracks were observed on the surface of metal and/or partial metal dentures, so the corrosion resistance decreases essentially. And in case, the duration is over than  $10 \mu\text{s}$ , the non-homogeneous surface could be obtained. This relation is summarized as

$$\tau \approx k \cdot r^2 / a, \quad (1)$$

where  $r$  is an extrapolated penetration depth of the electrons in material,  $a = \lambda \rho c$  is thermal diffusivity,  $\lambda$ ,  $\rho$ ,  $c$  are thermal conductivity, density, and heat capacity, respectively. For most of materials the value of  $a$  belongs to the range from 0.06 (for Ti) to  $1.12 \text{ cm}^2/\text{s}$  (for Cu). Concerning the value of  $r$ , it belongs to the range (at electrons energy 20-40 keV) from  $0.5\text{--}1.3 \mu\text{m}$  (for W) to  $3.3\text{--}9.3 \mu\text{m}$  (for Al), correspondingly.

The coefficient  $k$  depends on the material properties, namely it is defined by the relation between  $a$  and  $r$ . To provide a high efficiency of the surface heating, from one hand, and to decrease the thermal stresses defined by the temperature gradients in a surface layer from other hand, value of  $k$  is chosen by the following way:

- a) for most of constructive metallic alloys (alloys on the base of Fe, Al, and Ti), the coefficient  $k \approx 1\div 5$ .
- b) In case of the materials having high temperature conductivity and for small value of penetration depth  $r$  (Cu, Mo< W and alloys based on them) the coefficient  $k$  should be equal  $k \approx 10\div 50$ .

Using the Eq. 1 and taking into account the limitations of  $k$  one can obtain the range of the beam pulse duration  $\tau \approx 0.5\div 10 \mu\text{s}$

What is claimed is:

1. Pulsed Electron Beam System to use for the surface modification of the metal and/or partial metal dentures. The system is consisted with an explosive emission cathode, accelerating gap formed by the cathode and plasma anode, and an electron collector where the metal and/or partial metal dentures are fixed, which are placed into a guide magnetic field. The holder of the metal and/or partial metal dentures is made with metal materials offering cooling effect to the mentioned products.
2. The process to modify the surface of the metal and/or partial metal dentures using Pulsed Electron Beam Systems, the irradiated energy should be over than  $0.1 \text{ J/cm}^2$  and the pulse repetition is lower than 100.
3. The metal and/or partial metal dentures which surface is modified using pulsed electron beam irradiation for polishing from as-cast to finished and corrosion resistance modified products.